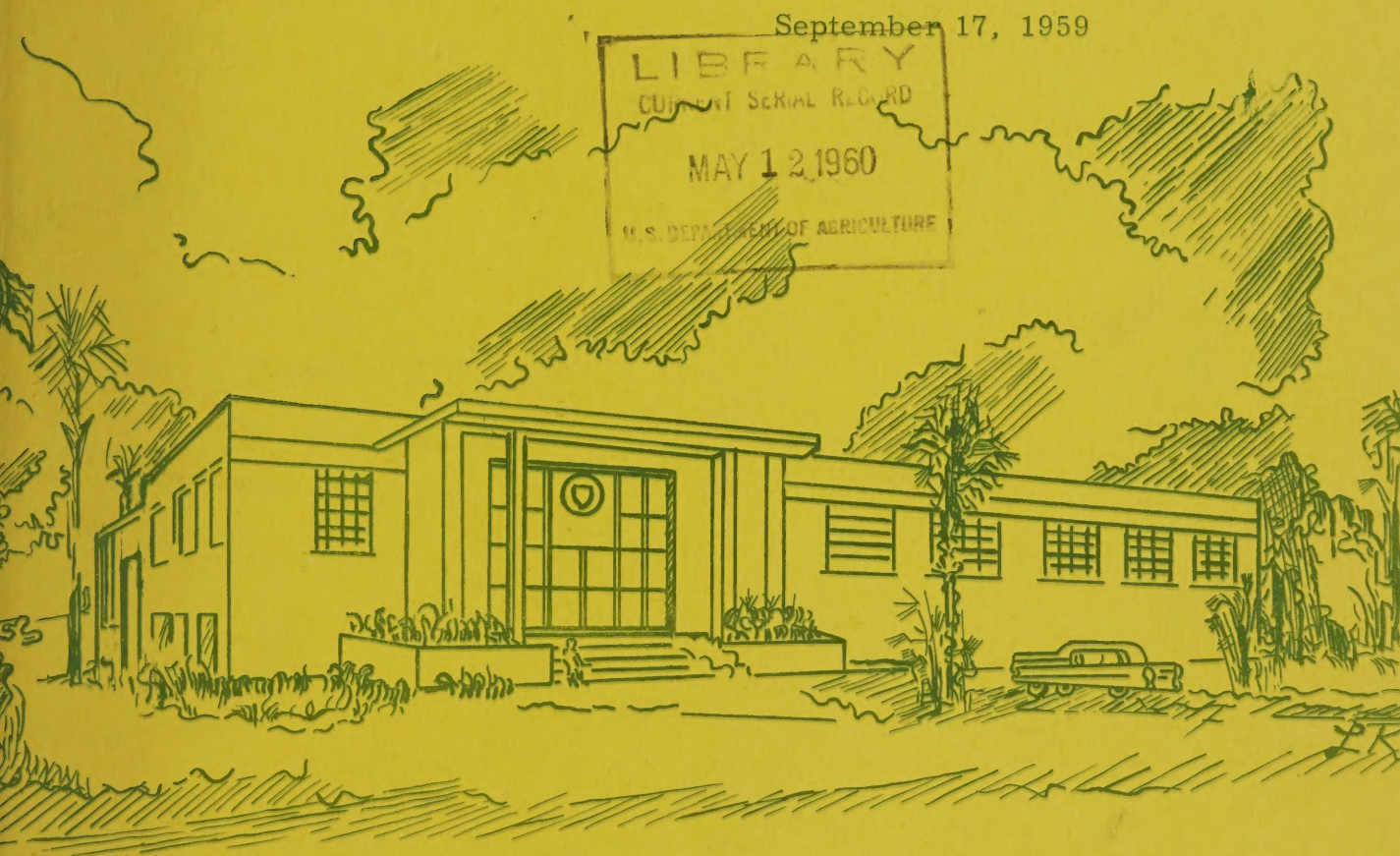
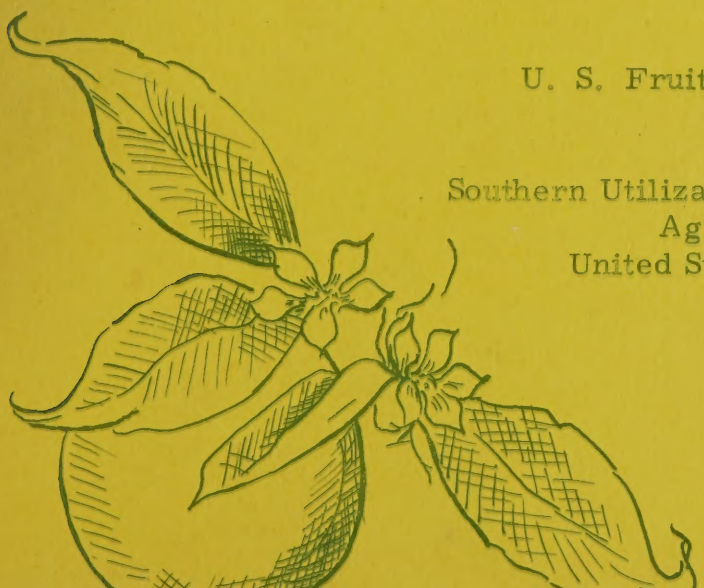
CITRUS

PROCESSING CONFERENCE

September 17, 1959





U. S. Fruit and Vegetable Products Laboratory
Winter Haven, Florida

Southern Utilization Research and Development Division
Agricultural Research Service
United States Department of Agriculture

PROGRAM AND ABSTRACTS OF PAPERS

NINTH CITRUS PROCESSING CONFERENCE

September 17, 1959

Florida Room, Citrus Building
Winter Haven, Florida

ORGANIZATIONS PARTICIPATING

IN

NINTH CITRUS PROCESSING CONFERENCE

SOUTHERN UTILIZATION RESEARCH AND DEVELOPMENT DIVISION

U. S. Fruit and Vegetable Products Laboratory, Winter Haven, Florida
U. S. Fruit and Vegetable Products Laboratory, Weslaco, Texas

WESTERN UTILIZATION RESEARCH AND DEVELOPMENT DIVISION

Fruit and Vegetable Chemistry Laboratory, Pasadena, California
Western Regional Research Laboratory, Albany, California

PROGRAM

CITRUS PROCESSING CONFERENCE
September 17, 1959

MORNING SESSION - 9:45 A. M.

(M. K. Veldhuis, In Charge, U. S. Fruit and Vegetable Products Laboratory, Winter Haven, Florida, Presiding)

OPENING REMARKS. M. K. Veldhuis

AFTERNOON SESSION - 1:30 P. M.

(V. H. McFarlane, Chief, Food Crops Laboratory, Southern Utilization Research and Development Division, New Orleans, Louisiana, Presiding)

ADDITIONAL CONTENTS

EMPIRICAL FACTORS FOR CONVERTING REFRACTOMETER AND DENSITY VALUES OF CITRUS JUICE PRODUCTS TO TRUE SOLUBLE SOLIDS

W. CLIFFORD SCOTT
U. S. Fruit and Vegetable Products Laboratory
Winter Haven, Florida

This study was undertaken to determine the possibility of developing empirical correction factors that could be used for converting refractometer, spindle, or pycnometer Brix values to true soluble solids with a reasonable degree of accuracy. True soluble solids were determined by vacuum drying an aliquot of supernatant centrifuged from single strength juice or diluted concentrate.

Twenty-four samples of single strength orange juice, 16 samples of single strength grapefruit juice, 15 samples of concentrated orange juice, and 12 samples of concentrated grapefruit juice were analyzed for °Brix by refractometer, hydrometer and/or pycnometer, percent soluble solids and total solids by vacuum drying, and titratable acidity. Most of the samples were obtained directly from processing plants over a period of several seasons in an effort to have them represent as wide a range of juices as is ordinarily encountered in control laboratories. It is anticipated that results reported here would be applicable throughout the industry.

Degrees Brix as determined by the refractometer, hydrometer, and pycnometer were compared to soluble solids determined by vacuum drying, with results as follows:

SINGLE STRENGTH ORANGE JUICE. (1) Refractometer. The average ratio of percent soluble solids to °Brix by the refractometer was 0.995 to 1 with a standard deviation of 0.014. In terms of juices with refractometer values of 12.0° Brix, two-thirds of the soluble solids values were within the range of 11.94 ± 0.17%. Maximum variation from the average was -0.48% soluble solids.

(2) Hydrometer. The average ratio of percent soluble solids to °Brix by the hydrometer was 0.969 to 1, with a standard deviation of 0.013. In terms of juices with hydrometer values of 12.0° Brix, two-thirds of the soluble solids values were within the range of 11.63 ± 0.16%. Maximum variation from the average was -0.31% soluble solids.

(3) Pycnometer. The average ratio of percent soluble solids to °Brix by the pycnometer was 0.956 to 1, with a standard deviation of 0.011. In terms of juices with pycnometer values of 12.0° Brix, two-thirds of the soluble solids values were within the range of 11.47 ± 0.13%. Maximum variation from the average was +0.29% soluble solids.

Similar results for single strength grapefruit juice and both orange and grapefruit concentrates are presented in the following table:

		Std.Dev.	Max.Dev.
SINGLE STRENGTH GRAPEFRUIT JUICE			
Sol. Solids/°Brix by Refractometer Ratio	.992	.009	.022
Sol. Solids in 12.0° Brix juices	11.90 %	±.11 %	+.26 %
Sol. Solids/°Brix by Hydrometer Ratio	.967	.015	.030
Sol. Solids in 12.0° Brix juices	11.60 %	±.18 %	-.36 %
Sol. Solids/°Brix by Pycnometer Ratio	.960	.011	.023
Sol. Solids in 12.0° Brix juices	11.52 %	±.13%	-.28 %
CONCENTRATED ORANGE JUICE			
Sol. Solids/°Brix by Refractometer Ratio	.984	.007	.012
Sol. Solids in 42.0° Brix concentrates	41.33 %	±.29 %	-.50%
Sol. Solids/°Brix by Pycnometer Ratio	.971	.010	.019
Sol. Solids in 42.0° Brix concentrates	40.78 %	±.42 %	+.80 %
CONCENTRATED GRAPEFRUIT JUICE			
Sol. Solids/°Brix by Refractometer Ratio	1.000	.005	.010
Sol. Solids in 42.0° Brix concentrates	42.00 %	±.21 %	-.42 %
Sol. Solids/°Brix by Pycnometer Ratio	.985	.013	.020
Sol. Solids in 42.0° Brix concentrates	41.37 %	±.55 %	-.84 %

Attention is called to the fact that refractometer values are higher than the true soluble solids for each type of product, except concentrated grapefruit juice. In those instances where refractometer values are taken for the purpose of determining true soluble solids contents of juices or concentrates, addition of corrections for citric acid only serve to compound the error already present in the method. According to results of this study, considerable improvement in accuracy can be obtained by judicious use of conversion factors.

These data indicate that for estimation of soluble solids in concentrated grapefruit juice, the average of a number of refractometric determinations will yield results equal to those obtained by vacuum drying, but that individual determinations may vary from true soluble solids by as much as 0.42° Brix. For orange concentrates, the average of a number of refractometric determinations will yield results about 0.7% too high. Upon multiplying the refractometer value of 42.0° Brix by the factor 0.984, the probable error would be reduced in two-thirds of the determinations to less than 0.3% solids, with the possible error being as great as 0.5° Brix.

VOLATILE BITTER SUBSTANCES IN ORANGE PEEL

LYLE J. SWIFT
U. S. Fruit and Vegetable Products Laboratory
Winter Haven, Florida

Bitter flavors in orange peel are of interest because of possible effects on flavors of juice and other citrus products. In a previous study, variations in soluble solids, acidity, pH, Brix-acid ratio, sugars soluble pectic substances, ascorbic acid, flavonoids, diacetyl, viscosity, color, specific gravity, and fluorescence were reported. It was observed that approximately 3% of peel juice was detectable in reconstituted orange juice. It was also observed that much of the bitter material in orange peel was volatile and could be distilled with steam. In addition, part of the bitter flavor is undoubtedly due to flavonoids. The current discussion is concerned primarily with compounds isolated from the volatile fraction. As before, effluent from a peel oil centrifugal was used as a source of material.

Small quantities of volatile fractions from filtered peel juice were subjected to analysis after recovery by steam distillation, extraction of the distillate with petroleum ether and evaporation of the solvent. Approximately 0.11 ml. of volatile organic material was recovered per liter of peel juice. Gas-liquid chromatographic (GLC) analyses yielded charts having a number of very small peaks (probably representing hydrocarbons) well separated from two prominent peaks which followed. By condensing the effluent gases from several GLC runs it was possible to accumulate enough of the compounds responsible for the large peaks to obtain infrared curves for them. These curves showed the first principal peak to be due to linaloöl and the second to be due to α-terpineol. Direct weights of these fractions indicated that there were about 23% linaloöl and 21% α-terpineol in the original volatile extract. Linaloöl and α-terpineol are constituents of normal peel oils, together making about 1% of the total oil.

In order to study the occurrence and effects of these alcohols, (linaloöl and α-terpineol), a method of estimating them is necessary. A good cold-pressed orange oil on hand showed only a very small peak for linaloöl and none for α-terpineol. Graduated amounts of linaloöl and α-terpineol were added, GLC tracings obtained, and a calibration curve constructed by plotting the amounts of constituents added against peak areas after correcting for the linaloöl already present. This method permitted the estimation of these alcohols in extracts at hand.

Studies have been conducted on the recovery of the alcohols from juice by distillation as a preliminary step to GLC analysis. Heat and acid have been reported as capable of changing limonene to terpineol. This was supported by an experiment in which added peel oil was distilled

from orange juice that had been previously freed of volatile oils by exhaustive distillation. This procedure was adopted to retain the original effects of acidity and buffering action. Results were as follows:

	mg.per ml.	
	linaloöl	α-terpineol
Original coldpressed peel oil	8.02	none
Same, distilled, distillate extract not added	4.66	2.00
Same, distilled, distillate extract added	5.33	9.33

This indicates that part of the linaloöl is lost (possibly by isomerism to geraniol) during distillation from acid solution and that terpineol is produced. Repetition of the experiment with limonene instead of peel oil also gave terpineol, indicating that it came from limonene.

Neutralization of orange juice before distillation was tried in an effort to decrease the rate of change to these alcohols. This procedure, followed by addition of distillate extract to the recovered oil, led to values of 0.44 mg. linaloöl and 0.13 mg. α-terpineol per liter of juice in contrast to comparable values of 0.50 and 1.50 mg. per liter, respectively, obtained without neutralization. Thus neutralization prevents terpineol formation to a great extent but doesn't appear to decrease the yield of linaloöl. Recoveries of linaloöl and α-terpineol added to juice were 25-29% and 63-76%, respectively, so recourse to empirical methods of analysis may be necessary.

The peel juice extract mentioned at the beginning of the abstract as containing about 23% linaloöl and 21% α-terpineol was obtained by distillation from acid juice and might be expected to show a fictitiously high terpineol value. An extract from neutralized peel juice gave about the same yield (0.45 ml. from 4 liters juice) and the percentage of linaloöl was not changed appreciably, but the α-terpineol was only about 8%.

At present, estimation of these substances in an isolated extract is reasonably satisfactory. The remaining problem is to arrive at a quantitative method, or at least a satisfactory empirical relationship, that will permit determination of linaloöl and α-terpineol in aqueous mixtures.

An estimate of that part of the bitterness of peel juice due to these substances was obtained by taste panel evaluations. By comparison of taste thresholds of peel juice with linaloöl and α-terpineol added to orange juice, it appears that these substances may be responsible for about one-third of the peel juice bitterness and contribute about equally to it. It was determined that as little as 7.6 mg. linaloöl and 2.5 mg. α-terpineol are detectable when present individually in one liter of orange juice.

RECENT DEVELOPMENTS IN THE CHEMISTRY OF CITRUS OILS

R. M. IKEDA, S. H. VANNIER AND L. A. ROLLE
Presented by
WILLIAM L. STANLEY
Fruit and Vegetable Chemistry Laboratory
Pasadena, California

Work has continued on the isolation and identification of coumarin compounds in lemon oil. Of the ten compounds isolated, nine have now been identified, the ninth one (most recently studied) being oxypeucedanin hydrate. Identity was based on carbon and hydrogen analysis, comparison of melting point with that reported in the literature, ultraviolet spectra and preparation of the diacetate.

The investigation of the aldehydes in lemon oil has extended the list from the seven compounds reported last year (C_8, C_9, C_{10}, C_{11} normal saturated aldehydes, citronellal, geranial and neral) to 14 compounds. All the normal saturated aldehydes from C_7 to C_{17} have been identified.

A quantitative method of analysis for the individual aldehydes in major concentration in citrus oils has been developed. The method is based on the recovery of the aldehydes as water-soluble Girard-T derivatives, regeneration with formaldehyde and gas chromatographic analysis. Optimum conditions for the method were worked out with a known mixture of aldehydes. Approximately 90% of the carbonyl components in lemon oil are accounted for in determining the content of C_7 to C_{11} normal saturated aldehydes and geranial and neral. Aldehyde analyses are presented for lemon, orange, grapefruit and lime oils.

A study has been conducted on the hydrocarbons in lemon oil samples collected over a period of two years. The mixture of hydrocarbons was separated from other components in the oils by downward elution on chromatostrips. The eluate without concentration was analyzed by gas chromatography.

Four hydrocarbons were found to be in major concentration: α-pinene, β-pinene, d-limonene and an unknown compound. Camphene was found in traces as well as several other unknowns. Para-cymene was found in badly deteriorated oils in which the color of the oil had noticeably faded. Of the hydrocarbon mixture limonene varied from 60 to 70%. β-pinene concentration was about 10 times that of α-pinene.

RECENT DEVELOPMENTS IN THE CHEMISTRY OF CITRUS FLAVONOIDS

ROBERT M. HOROWITZ AND BRUNO GENTILI
Presented by
E. A. BEAVENS
Fruit and Vegetable Chemistry Laboratory
Pasadena, California

Work on the isolation and proof of structure of the lemon flavonoids has continued. At the present time the following flavonoids have been isolated and identified: hesperidin, eriodictyol (and its glycoside), quercetin, isorhamnetin, limocitrin A (and its glycoside), limocitrin B (and its glycoside), apigenin, luteolin, diosmin and chrysoeriol. In addition, the following related compounds have been obtained: scopoletin, umbelliferone, sinapic acid and p-coumaric acid. A number of these have been made available to the Pharmacology Laboratory at Western Utilization Research and Development Division for metabolic studies.

Eriodictyol glycoside has been isolated from lemons as an amorphous powder, which appears to be essentially homogeneous on paper chromatograms. It yields a crystalline acetyl derivative. Methylation and spectroscopic studies have shown that the structure of this glycoside is eriodictyol 7-β-rutinoside. Hesperidin has an identical structure except for the presence of a methyl group on the 4'-hydroxyl. Szent-Györgyi's unsupported statement (published in 1936), that lemons contain a glycoside of eriodictyol, the structure of which is demethylated hesperidin, is therefore correct.

The relationship between bitterness and chemical structure of flavonoid glycosides is being studied. Naringin, which has long been recognized as the chief bitter constituent of grapefruit, is generally considered to be the 7-rutinoside of the aglycone naringenin. Neohesperidin, one of the principal flavonoids of the bitter orange, is isomeric with hesperidin and differs from it only in the point of attachment of rhamnose to glucose. We have now found that both naringin and neohesperidin undergo cleavage in alkali to yield, in each case, the same phloracetophenone rhamnoglucoside:

$$\text{Naringin } \underline{\text{or}} \text{ Neohesperidin} \xrightarrow{\text{NaOH}} \text{Rhamnose-Glucose-Phloracetophenone}$$

It is of interest that both neohesperidin and the phloracetophenone rhamnoglucoside are bitter, though not to the same degree as naringin.

The significance of these findings is as follows: (1) Naringin is <u>not</u> a rutinoside and this probably accounts for the puzzling fact that its solubility and taste differ markedly from those of hesperidin. (2) The disaccharide of both naringin and neohesperidin is identical. It is a rhamnoglucose (called "neohesperidose") and is isomeric with rutinose, the only other known rhamnoglucose. (3) The presence of neohesperidose in flavanone glycosides is intimately associated with the property of bitterness.

Methylation of naringin followed by hydrolysis and identification of the methylated sugars has shown that in neohesperidose, rhamnose is attached to the 2-hydroxyl group of glucose. Disaccharides attached through the 2-hydroxyl are of relatively rare occurrence.

The limitations of the Davis method in estimating citrus flavanones will be discussed.

REPORT OF PROGRESS ON CITRUS RESEARCH AT THE U. S. FRUIT AND VEGETABLE PRODUCTS LABORATORY, WESLACO, TEXAS

FRANCIS P. GRIFFITHS
U. S. Fruit and Vegetable Products Laboratory
Weslaco, Texas

Major work on citrus at the Weslaco laboratory during the past year has been on effect of pulp fortification of late season grapefruit juice and appraisal of pulp fortified versus unfortified juice from colored grapefruit; production and keeping quality of chilled single strength grapefruit juice and grapefruit-orange juice blends; development of better analytical methods for the estimation of naringin; and, in cooperation with California Institute of Technology, the mechanism of carotogenesis in grapefruit and tomatoes. Cooperative studies with Dr. Cooper of the Citrus Rootstock Investigations Laboratory on the effect of different rootstocks on the processing quality of Ruby Red grapefruit were concluded at the end of the 1958-59 citrus season.

A series of in-plant experiments was run on pulp fortification using standard equipment of a large citrus juice processor. Early in the season, December, 1958, excess pulp from grapefruit was collected from paddle finishers and after addition of one quart of juice to each gallon of pulp, the pulp was passed through a Fitzpatrick comminuting mill equipped with a .040 inch screen. After deaeration the pulp was pasteurized at 195° F. and filled into 46 ounce cans, cooled and stored. This pulp retained excellent color during storage. After 3-1/2 and 4-1/2 months it was used to color fortify late season juice of low color characteristics. Color of the juice was improved by the addition of approximately 10% of the stored pulp. A taste panel was unable to consistently distinguish taste differences between the pulp fortified and unfortified juices. Table 1 summarizes this information.

Table 1. Effect of addition of stored pulp on qualities of late season poorly colored red grapefruit juice.

	Acid %	Brix °	Suspended Solids %	Naringin %	Color 1/
Original pulp	0.98	8.5-9.5	84-100	0.170	+ .929
Pulp after 3-1/2 mo. storage	1.02	9.5	83	0.228	+1.083
March juice, control	1.15	9.0	8	0.091	- .110
March juice, pulp-fortified	1.18	9.2	10	0.098	+ .061
April juice, control	1.00	10.0	8.5	0.083	- .211
April juice, pulp fortified	1.03	10.0	12.5	0.096	+ .031

1/ Color is expressed as a/b from the a and b readings of the Gardner Automatic Color Difference Meter. + readings are considered acceptable.

It is concluded that the use of early season canned pulp to add color to late season grapefruit juice is a feasible procedure for standardizing the color of canned Ruby Red grapefruit juice.

Two seasons work on the production and keeping quality of chilled single strength Texas grapefruit juice, and one season on Texas orange juice, and grapefruit-orange juice blends, have been completed. Grapefruit juice stored in glass containers to more closely simulate bulk storage conditions kept 14 days at 40° F. or below. The shelf life of grapefruit juice could be extended by flash pasteurization at 165°, 170°, 175° or 180° F. without adversely affecting flavor. Flash pasteurization at 180° F. enabled grapefruit juice to be stored 21 days at 32° F. before adverse flavor changes occurred. Of nine packs pasteurized at 180° F. and kept at 32° F. for 28 days, two packs developed unacceptable flavor changes. Unstabilized grapefruit-orange juice blends and orange juice controls were unsatisfactory after 14 days storage at all storage temperatures, 32°, 40°, 50° F., either because of cloud loss or spoilage. Flash pasteurization at 180° F. of grapefruit-orange juice blends and plain orange juice did not adversely affect flavor. Stabilized (180° F.) blended juice and orange juice retained satisfactory quality for 21 days at 32° F., but occasional packs showed flavor deterioration to an unsatisfactory level after 28 days at 32° F. Marked deterioration occurred in 21 days at 40° F. At 50° storage all packs were spoiled before the 14th day of storage.

Little decrease in vitamin C content occurred during juice storage, losses averaged 7% for grapefruit and 13% for orange juice. The greatest loss occurred during the first two weeks of storage.

Seasonal changes in juice composition did not significantly affect keeping quality. Storage temperatures affected storage life of heat stabilized juices more than did variations in the pasteurization temperatures.

Column chromatography of grapefruit juice using 1 part magnesol and 2 parts by weight of celite as the adsorbent has enabled the separation of naringin from sugars and fruit acids. Naringin can be removed from the column by dilute alcohol. Approximately 10 percent of the Davis value of the original juice is due to a non-naringin component not retained on the column. Work is continuing in an effort to develop a better analytical procedure for naringin.

Four seasons cooperative investigation with the Rootstocks Investigations Laboratory, ARS, on the effect of different rootstocks on the processing quality of Ruby Red grapefruit have been completed. Analysis of these data are incomplete.

Fundamental investigations on the synthesis of carotene and lycopene by Ruby Red grapefruit and tomatoes are in progress at California Institute of Technology by Dr. Purcell of the Weslaco laboratory, and Dr. Bonner

and his associates. Mevalonic acid has been shown to be converted to carotenoids in the tomato fruit. Cross fruit grafting experiments - white fruit on a Ruby Red tree, and Ruby Red fruit on a white grapefruit producing tree - demonstrated that the carotenes are not translocated and the site of color synthesis is in the growing fruit and not in the stem or leaf of the tree. White fruit grafted on a Ruby Red producing tree remained white, whereas Ruby Red grapefruit grafted while immature, (2 inch dia.) onto a white grapefruit tree, continued to produce color.

PRELIMINARY REPORT OF INVESTIGATIONS ON STABILITY OF CITRUS SALADS

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Winter Haven, Florida

Chilled citrus salad is a highly perishable item of commerce which depends for marketability on the prevention of microbial growth during distribution. Primary dependence is placed upon the maintenance of low temperature, although sodium benzoate may be added to further slow microbial growth.

An indication of the temperature ranges to which chilled citrus salads may be subjected was obtained by placing recording thermometers in close proximity to citrus salad displays in local markets. Temperatures were recorded within a range from 40°F. to 60°F. A series of commercial packs of mixed orange and grapefruit sections with and without added benzoate were exposed to temperatures in this range and rates of microbial growth determined. In one series, off-flavor development was observed in 4 days at 60°F., in 8 days at 50°F., and in 43 days at 40°F. Sodium benzoate, in this series, was determined to be 0.044% in the cover syrup and the pH was 4.2. Under these conditions microbial growth was appreciably inhibited at the lower temperatures by benzoate, although off-flavor was not retarded. In certain other series of packs spoilage was significantly retarded at the lower temperatures but not at 60°F. or 70°F.

Representative colonies were picked from the plates and screened for off-odor development in mixed orange-grapefruit juice. These have been preserved for further investigation.

The effect of sodium benzoate at several levels and pH values on representative spoilage organisms is being determined. A similar series of experiments is planned with other preservatives.

REVIEW OF WORK OF WESTERN UTILIZATION RESEARCH AND DEVELOPMENT DIVISION ON ORANGE JUICE POWDER

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Western Regional Research Laboratory
Albany, California

Investigations of various research workers to develop citrus juice powders extend over at least the past 30 or 40 years. Spray drying of citrus juices containing high percentages of drying aids was under way prior to World War II. A lemonade powder containing 20% lemon juice solids and 80% corn sirup solids was used in appreciable quantities by the Military during World War II. During this period research was carried out that led to the construction of a commercial size plant for the vacuum drying of 100% orange juice powder. The end of the war, with the resulting cancellation of contracts, and the advent of frozen orange juice concentrate caused the conversion of this plant to a concentrate operation.

Interest in orange juice powder decreased until the work at the Western Utilization Research and Development Division brought some new approaches which promised a better product with greater storage life. The work eventually brought together increased knowledge regarding frozen concentrate for raw material, flavor fortification by means of orange oil, in-package desiccation by special types of calcium oxide, and the use of continuous vacuum belt drying as developed by the Chain Belt Company.

Our first approach to the production of citrus powders involved the use of drying aids. Both spray and vacuum drying were tried but spray drying was eventually given up because of losses during drying and relatively low product quality. Vacuum drying was done in a shelf type dryer using pressures down to 1 mm. Low conversion corn sirup solids gave the best improvement in ease of drying and handling and increased storage stability. The addition of sulfur dioxide up to 250 ppm was found to permit use of higher drying temperatures and to increase storage stability.

Work at our Pasadena Laboratory had shown that flavor could be returned to frozen concentrate by addition of oil. Following this lead flavor fortification was tried by two methods: addition of oil to the concentrate prior to drying, and addition of oil to the product after the oil had been entrapped in sorbitol granules. The latter gave the better results.

In-package desiccation was found to be another improvement to increase storage stability. By use of calcium oxide the moisture content of orange powders can be reduced from 3-4% to below 1%, which greatly improves storage stability under adverse conditions.

The Quartermaster Food and Container Institute became interested in orange juice powder from samples we submitted, and in 1952 contracted with Vacu-dry Company for production of a limited quantity of powder containing 40% corn sirup solids. This was successfully produced in batch vacuum dryers.

Concurrently with the laboratory work mentioned above, work was also being done on orange juice powder with no drying aid. With proper choice of raw materials, it was found that an excellent product could be produced that had acceptable storage life. To increase the commercial feasibility of this product, continuous drying seemed desirable. Arrangements were made with Chain Belt Company to make runs on their experimental continuous vacuum belt dehydrator. Vacu-dry Company participated in the first part of these runs. Although corn sirup solids facilitated the continuous drying operations, it was shown that straight orange juice powder could be run successfully. The Quartermaster Food and Container Institute subsequently contracted with Vacu-dry Company for production of a limited quantity of straight orange juice powder on the Chain Belt experimental dehydrator.

Further research is needed to improve the commercial practicality of the production of vacuum dried orange powder.

SOME POSSIBLE CAUSES OF CITRUS OXIDIZED FLAVOR

M. K. VELDHUIS
U. S. Fruit and Vegetable Products Laboratory
Winter Haven, Florida

Citrus oxidized flavor, otherwise characterized as cardboard off-flavor, castor oil flavor, or merely "COF", has been observed in frozen concentrated citrus juices occasionally to a pronounced degree and more frequently to a lesser extent. Generally, it has not been observed in freshly prepared frozen concentrate, but has developed after a month or two of storage at 0°F. or below. After a few additional months of storage, the off-flavor decreased and was frequently difficult to detect. The number of cases in which a very pronounced oxidized flavor was found has not been sufficiently great to be of economic importance, but the increased number of cases where it may be at a barely detectable level and is associated with a weakness in flavor, makes it much more important.

One method of control has been to maintain peel oil at a fairly high level in concentrates, but whether this contributes by masking the flavor, controlling the formation of the responsible compounds, or both is not clear. Even this method is only partially successful.

"COF" has been characterized as resembling rancid fat in flavor and aroma. In some cases the juice seemed to have a "slippery" feel or tallowiness in the mouth.

Dr. J. S. Blair et al. [Food Technol., 11, 61-8 (1957)] have described the phenomenon and postulated a relationship with physiological immaturity. They claim to have simulated tallowiness by dispersing in orange juice the higher fatty acids obtained by saponification of debittered grapefruit seed oil. They believe the malodor to be correlated with odorous components, disagreeable at times, which characterize unrefined vegetable oils in general. They believe that α-, β- unsaturated aldehydes constitute a part of the malodor and obtain weak Hiltner-Wearn tests for such constituents.

It is possible, even likely, that there are a number of compounds involved in "COF". When an opportunity occurred recently to observe the odor of a number of compounds isolated from peel oil, a resemblance was noted between the aroma of some straight chain aldehydes and "COF". The resemblance was most striking with nonanal and decanal. Somewhat similar aromas were observed with octanal and undecanal but they were not considered as typical. Small quantities of these compounds were obtained and dispersed in orange juice where the same results were observed. Much of the flavor and aroma was duplicated; the tallowiness, however, was not simulated. Normal saturated aldehydes have been found in citrus juices. Kirchner and Miller [J. Agr. Food Chem., 5, 283-91 (1957)] found hexanal, octanal, and decanal in fresh and freshly canned orange

juice, but only a trace of decanal in stored juice. 2-Dodecenal was listed as possibly present in fresh and freshly canned juice, but not in stored juice.

When n-decanal and n-nonanal were added to frozen concentrated orange juice and the product stored at 40°F. and 0°F., characteristic "COF" aromas and flavors decreased markedly during storage in a few weeks at 40°F. and in a few months at 0°F. As the original "COF" odor decreased, another less potent odor appeared which may have been due to formation of acids from aldehydes by oxidation.

Proof that these aldehydes contribute to "COF" is not complete at present and it may be very difficult to establish definite association. Aldehydes are quite unstable and susceptible to mild oxidation and other reactions.

Investigations are proceeding using distillation, extraction, and gas chromatographic techniques. Information would be appreciated whenever "COF" is encountered, especially if severe. Material of this nature is needed for investigation.

LIST OF CITRUS PUBLICATIONS

(September 1, 1958 - August 31, 1959)

UNITED STATES DEPARTMENT OF AGRICULTURE
AGRICULTURAL RESEARCH SERVICE

SOUTHERN UTILIZATION RESEARCH AND DEVELOPMENT DIVISION

U. S. FRUIT AND VEGETABLE PRODUCTS LABORATORY
600 Avenue S, N. W.
Winter Haven, Florida

PROCESSING FREEZE DAMAGED ORANGES
Bissett, O. W.
Proc. Florida State Hort. Soc., 71, 244 - 59 (1958).

U. S. FRUIT AND VEGETABLE PRODUCTS LABORATORY
509 West 4th St.
Weslaco, Texas

THE INCORPORATION OF MEVALONIC ACID INTO TOMATO CAROTENOIDS
Purcell, A. E., Thompson,*G., Jr., and Bonner,*J.
J. Biol. Chem., 234, 1081 - 4 (1959).

PIGMENTATION, PIGMENT ANALYSES AND PROCESSING OF COLORED GRAPEFRUIT
Lime, B. J., Stephens, T. S., Purcell, A. E., and Griffiths, F. P.
J. Rio Grande Valley Hort. Soc., 13, 30 - 8 (1959).

CAROTENOID PIGMENT FORMATION IN COLORED GRAPEFRUIT
Purcell, A. E.
J. Rio Grande Valley Hort. Soc., 13, 39 - 44 (1959).

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